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(54) **PROCESS FOR THE PREPARATION OF ELECTROSTATICALLY CHARGED PARTICLES**  
**VERFAHREN ZUR HERSTELLUNG VON ELEKTROSTATISCH GELADENEN TEILCHEN**  
**PROCEDE DE PREPARATION DE PARTICULES CHARGEES DE MANIERE ELECTROSTATIQUE**

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**EP-A- 0 234 841** **EP-A- 0 260 638**  
**EP-A- 0 493 076** **GB-A- 2 272 434**

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**EP 0 769 031 B1**

## Description

The present invention relates to a process for the preparation of electrostatically charged particles and, in particular, to the preparation of electrostatically pre-charged powders of high electrical resistivity such as powder paint, household cleaning particles and charged particles for biomedical applications.

Electrostatic coating techniques are increasingly used in commercial applications where durable, high quality, finishes are required. Electrostatic coating techniques involve the application, usually by spraying, of electrostatically charged particles, which preferably are pigmented, onto a substrate, generally but not exclusively a metal substrate, followed by baking to form a uniform and adherent coating. The electrical charge of the pigment-containing paint particles causes the attraction of the particles onto the substrate and holds them in place until the heat treatment is applied. Electrostatic coating techniques are used almost exclusively in the domestic white-goods market, and increasingly in the automobile industry. Less well known areas of application include the furnishing industry and bottle coating applications.

There are two known processes for electrostatically charging paint particles which are by corona charging or by tribo or frictional effects. Both of these techniques are known to be inefficient and extremely unpredictable. The important process of charging the powder is the responsibility of the end user and is usually accomplished by means of commercially available equipment.

The corona charging process relies on the creation of a high charge density unipolar ionic cloud, the ions of which subsequently attach onto the outer surface of discreet particles which are dispersed into an airborne cloud. Typically, only about 0.5% of all the charged ions become attached to the airborne particles, the remaining 95.5% remaining as free ions which can be extremely detrimental in coating applications to the quality of the coating.

Tribo or frictional charging of particles is unpredictable and the charging process is sensitive to a number of parameters including the nature of the materials used, relative humidity, duration of contact, relative permittivity, surface conditions, the type of contact and possibly electrical resistivity.

Accordingly, the prior art processes suffer from the disadvantage that the charging of the particles relies upon surface mechanism and the charging process is generally under the control of the end user who will have very little control over the level of charge deposited onto individual particles.

GB-A-2272434 discloses a method of importing a large second non-linearity into glasses by electron implantation, in which a suitable glass is exposed to an electron beam whereby a large fraction of the bombarding electrons penetrate into the glass sample where they get trapped and set up a space charge field.

We have now developed a more reproducible process for charging powder particles which overcomes the disadvantages of the prior art.

Accordingly, the present invention provides a process for the preparation of electrostatically charged particles of a high resistivity material which process comprises incorporating a unipolar charge into the material at a temperature at or above the glass transition temperature thereof or above the melting point thereof, the said unipolar charge being incorporated into the bulk of the material and the charged material being subsequently comminuted, or the said charge being incorporated into the material whilst forming particles thereof.

As is known by those skilled in the art the term glass transition temperature is used to define the temperature at which an amorphous material, such as a polymer or glass, changes from a brittle state to a plastic state.

The process of the present invention may be effected either by the incorporation of the unipolar charge into the material by the injection of unipolar charge or by the induction of unipolar charge whilst the material is at or above its glass transition temperature or above its melting point. The unipolar charge may be incorporated into the bulk of the material and particles thereof subsequently formed by comminution, for example by grinding. During the comminution process the material will retain its charge which is effectively locked into the material. Alternatively, the unipolar charge may be incorporated into the material during the formation of particles thereof, for example by extrusion through a nozzle of predetermined aperture. The particles produced in accordance with the present invention may be positively or negatively charged, as desired.

Examples of materials which may be charged according to the process of the present invention are polymeric materials such as polyethylene or epoxy/polyester, or wax materials such as a synthetic paraffin wax or a natural wax, for example Carnauba wax.

The charged particles produced in accordance with the process of the present invention preferably have a particle size in the range of from 10 to 300µm more preferably in the range of from 10 to 40µm.

The process of the present invention may be used to produce electrostatically charged particles of a composite polymeric material for use as a powder paint in coating applications, the composite polymer material generally incorporating one or more pigments therein. Other applications for the charged particles of the present invention are for use as household cleaning particles, as charged particles for biomedical purposes and charged particles for insect control purposes.

The present invention also includes within its scope electrostatically charged particles of a polymeric material or a wax wherein a unipolar charge is incorporated within the volume of the particles.

The charged particles of the present invention possess a more reproducible level of charge than was possible using the prior art charging techniques. In particu-

lar the advantages are that the electrical charge is located within the volume of the particles and the particles are pre-charged at the manufacturing stage, rather than being charged immediately prior to use. This results in the charge being retained for much longer than surface charged particles and also provides the particles with better sprayability characteristics than the surface charged particles.

The present invention will be further described with reference to the accompanying drawings, in which;

Figure 1 is a schematic drawing showing the electrical charge sited on the surface of the prior art paint particles;

Figure 2 is a schematic diagram showing the electrical charged sited within the pre-charged paint particles of the present invention;

Figure 3 is a schematic drawing of an apparatus for the induction charging of particles in accordance with the process of the present invention; and

Figure 4 is a schematic diagram of an apparatus for the corona injection charging of particles in accordance with the process of the invention.

Referring to the drawings, Figure 1 illustrates a paint particle 1 which has a negative charge 2 on the surface thereof. The bulk volume of the particle illustrated at 1a by an internal segment view of the particle is not charged. Figure 2 illustrates a pre-charged particle produced in accordance with the process of the present invention. The particle 3 does not have any charge on the surface thereof. A segment of the interior of the particle is illustrated at 4 where it is clearly shown that the bulk volume of the particle is negatively charged.

Although the particles 1 and 3 are shown with either a surface negative charge or a volume negative charge, respectively, it will be understood that the particles could equally well be provided with a positive unipolar charge.

The injection of the unipolar charge, either positive or negative charge, is achieved during the formation of the particles. Referring to Figure 3, this Figure illustrates schematically apparatus for the induction charging of particles, for example of paint, as they are formed. A nozzle through which the polymer is forced is illustrated schematically at 5. The particles 11 form as the material is forced through the nozzle. The particles are passed through an induction ring 6 which is required for the creation of the appropriate electrical field resulting from the application of a voltage by the generator 7. During passage of the particles 11 through the charged ring 6 the material accepts a unipolar charge and as the particles 11 subsequently harden the charge becomes effectively frozen within the volume of the particles.

An alternative method for imparting unipolar charge 2 particles is illustrated in Figure 4 in which the nozzle through which the molten material is extruded is shown schematically at 8. The material is extruded through the nozzle 8 and particles 11 are formed during this process.

A high voltage electrode 9 is incorporated within the nozzle and supplies a unipolar injection of charge into the bulk of the molten material as the beads 11 of the material are formed. The high voltage to the electrode 9 is supplied by the generator 10.

Charge injection either by high field injection, or by induction, may also be achieved by the incorporation of similar electrodes adjacent the final cooling roller systems in conventional extruders. Normally, the material will be produced in sheet form, to be subsequently ground into a fine powder.

The present invention will be further described with reference to the following Examples.

#### EXAMPLE 1

Unipolar charge injection into a molten example of an epoxy/polyester composite polymer was carried out using an electrode similar to that illustrated in Figure 4 of the accompanying drawings. The polymer sample size was 4.5cm in diameter and 0.6cm thick. The temperature was maintained at approximately 100°C, at which temperature the sample was above its glass transition temperature. Whilst at this temperature, a potentially -3KV at the sharp pointed electrode was maintained for a period of time during which the sample re-solidified. A initial charge value of  $-0.1 \times 10^{-9}$  was measured within the sample. The following day the charge value was the same and subsequently stabilised at approximately this value. The epoxy/polyester composite polymer sample was then subsequently comminuted.

#### EXAMPLE 2

Unipolar charge injection into a molten sample of Carnauba wax was carried out using an electrode arrangement similar to that illustrated in Figure 4 of the accompanying drawings. The wax sample size was 4.5cm in diameter and 0.6cm thick. The temperature was maintained at approximately 130°C at which the wax was molten. Whilst molten, a potential of 40 volts was maintained at the pointed electrode for a period of time corresponding to the time taken for the wax to re-solidify. An initial charge value of  $-0.3 \times 10^{-9}$  Coulombs was measured within the sample. The next day this value had decreased to  $-0.19 \times 10^{-9}$  Coulombs. This value was maintained over a period of weeks. The wax sample was subsequently comminuted.

#### Claims

1. A process for the preparation of electrostatically charged particles of a high resistivity material which process comprises incorporating a unipolar charge into the material at a temperature at or above the glass transition temperature thereof or above the melting point thereof, the said unipolar charge being

incorporated into the bulk of the material and the charged material being subsequently comminuted, or the said charge being incorporated into the material whilst forming particles thereof.

2. A process as claimed in claim 1 wherein the incorporation of unipolar charge is effected by the injection of unipolar ions into the material.
3. A process as claimed in claim 1 wherein the incorporation of unipolar charge is effected by the induction of unipolar ions into the material.
4. A process as claimed in any one of the preceding claims wherein the unipolar charge is incorporated into particles of the said material during the formation of the said particles by extrusion.
5. A process as claimed in any one of the preceding claims wherein the material is a polymeric material or a wax.
6. A process as claimed in any one of the preceding claims wherein the polymeric material is a polymer paint.
7. A process as claimed in claim 5 wherein the wax is synthetic paraffin wax or a natural wax.
8. A process as claimed in any one of the preceding claims wherein the electrostatically charged particles have an average particle size in the range of from 10 to 300  $\mu\text{m}$ .
9. Electrostatically charged particles of a polymeric material or a wax wherein a unipolar charge is incorporated within the volume of the particles.
10. Electrostatically charged particles as claimed in claim 9 wherein the polymeric material is a polymer paint.
11. Electrostatically charged particles as claimed in claim 9 wherein the wax is a synthetic paraffin wax or a natural wax.
12. Electrostatically charged particles as claimed in any one of claims 9 to 11 which have an average particle size in the range of from 10 to 300  $\mu\text{m}$ .
13. A powder paint which comprises electrostatically charged particles as claimed in any one of claims 10 to 12.

#### Patentansprüche

1. Verfahren zur Herstellung von elektrostatisch gela-

denen Teilchen eines Materials mit hohem spezifischen Widerstand, wobei das Verfahren umfaßt Inkorporieren einer unipolaren Ladung in das Material bei einer Temperatur bei oder oberhalb seiner Glas-Übergangstemperatur oder oberhalb seines Schmelzpunktes, wobei die unipolare Ladung in die Masse des Materials inkorporiert wird und das geladene Material anschließend zerkleinert wird oder die Ladung in das Material während der Teilchenbildung inkorporiert wird.

2. Verfahren nach Anspruch 1, worin das Inkorporieren unipolarer Ladung durch Injektion unipolarer Ionen in das Material bewirkt wird.
3. Verfahren nach Anspruch 1, worin das Inkorporieren unipolarer Ladung durch Induktion unipolarer Ionen in dem Material bewirkt wird.
4. Verfahren nach einem der vorhergehenden Ansprüche, worin die unipolare Ladung in Teilchen des Materials während der Bildung dieser Teilchen durch Extrusion inkorporiert wird.
5. Verfahren nach einem der vorhergehenden Ansprüche, worin das Material ein Polymerwerkstoff oder ein Wachs ist.
6. Verfahren nach einem der vorhergehenden Ansprüche, worin der Polymerwerkstoff eine Polymerfarbe ist.
7. Verfahren nach Anspruch 5, worin das Wachs ein synthetisches Paraffinwachs oder ein natürliches Wachs ist.
8. Verfahren nach einem der vorhergehenden Ansprüche, worin die elektrostatisch geladenen Teilchen eine durchschnittliche Teilchengröße im Bereich von 10 bis 300  $\mu\text{m}$  aufweisen.
9. Elektrostatisch geladene Teilchen aus einem Polymerwerkstoff oder einem Wachs, worin eine unipolare Ladung innerhalb des Volumens der Teilchen inkorporiert ist.
10. Elektrostatisch geladene Teilchen nach Anspruch 9, worin der Polymerwerkstoff eine Polymerfarbe ist.
11. Elektrostatisch geladene Teilchen nach Anspruch 9, worin das Wachs ein synthetisches Paraffinwachs oder ein natürliches Wachs ist.
12. Elektrostatisch geladene Teilchen nach einem der Ansprüche 9 bis 11, die eine durchschnittliche Teilchengröße im Bereich von 10 bis 300  $\mu\text{m}$  aufweisen.

13. Pulver-Farbe, die elektrostatisch geladene Teilchen nach einem der Ansprüche 10 bis 12 umfaßt.

10. Particules chargées de façon électrostatique selon la revendication 9, dans lesquelles le matériau polymérique est une peinture polymère.

#### Revendications

1. Procédé de préparation de particules chargées de façon électrostatique constituées d'un matériau à résistivité élevée lequel procédé comprend l'incorporation d'une charge à polarité unique à l'intérieur du matériau à une température se situant au niveau ou au-dessus de la température de transition vitreuse de celui-ci ou au-dessus du point de fusion de celui-ci, ladite charge à polarité unique étant incorporée à l'intérieur de la masse du matériau et le matériau chargé étant ensuite réduit en poudre, ou bien ladite charge étant incorporée à l'intérieur du matériau tout en formant des particules de celui-ci.

2. Procédé selon la revendication 1, dans lequel l'incorporation d'une charge à polarité unique est réalisée par l'injection d'ions à polarité unique à l'intérieur du matériau.

3. Procédé selon la revendication 1, dans lequel l'incorporation d'une charge à polarité unique est réalisée par l'induction d'ions à polarité unique à l'intérieur du matériau.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel la charge à polarité unique est incorporée à l'intérieur de particules dudit matériau durant la formation desdites particules par extrusion.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel le matériau est un matériau polymère ou une cire.

6. Procédé selon l'une quelconque des revendications précédentes, dans lequel le matériau polymère est une peinture polymère.

7. Procédé selon la revendication 5, dans lequel la cire est une cire paraffinique synthétique ou une cire naturelle.

8. Procédé selon l'une quelconque des revendications précédentes, dans lequel les particules chargées de façon électrostatique présentent une taille de particule moyenne dans la plage allant de 10 à 300 µm.

9. Particules chargées de façon électrostatique d'un matériau polymérique ou d'une cire dans lesquelles une charge à polarité unique est incorporée à l'intérieur du volume des particules.

5 11. Particules chargées de façon électrostatique selon la revendication 9, dans lesquelles la cire est une cire paraffinique synthétique ou une cire naturelle.

10 12. Particules chargées de façon électrostatique selon l'une quelconque des revendications 9 à 11, qui présentent une taille de particule moyenne dans la plage allant de 10 à 300 µm.

15 13. Peinture en poudre qui comprend des particules chargées de façon électrostatique selon l'une quelconque des revendications 10 à 12.

FIG. 1.

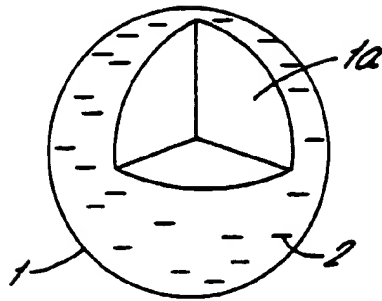


FIG. 2.

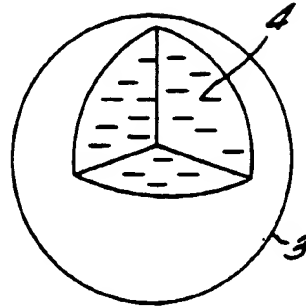


FIG. 3.

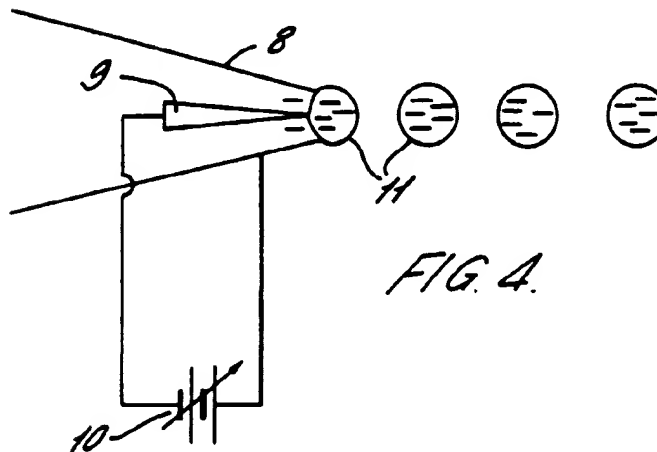
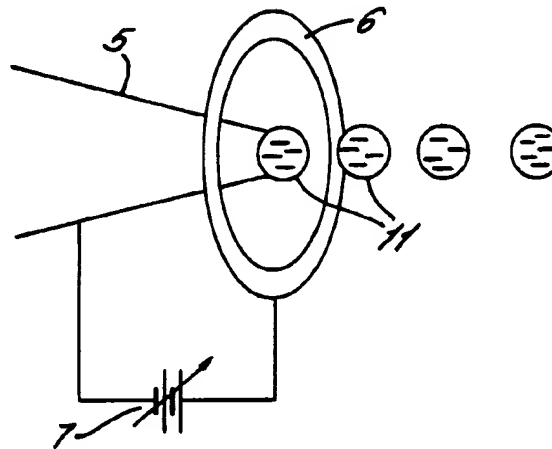


FIG. 4.